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Rock Island Arsenal Laboratory

TECHNICAL REPORT

IMPROVING THE RUST PREVENTIVE PROPERTIES OF MULTIPURPOSE GREASES

By

R. L. Young

Dept. of the Army Project No. 1-A-0-24401-A-107

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31 January 1963

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Rock Island Arsenal
Rock Island, Illinois

ABSTRACT

Eighteen compounds or mixtures were selected for testing as rust preventive additives for greases. They were intimately mixed into a selected base grease in predetermined percentages. A screening test was developed and those additives showing up well on it were further tested by Method 4012 of Federal Standard No. 791. The greases containing the best performing additives were checked to determine if any of the requirements of MIL-G-10924B, Grease, Automotive and Artillery, had been seriously altered. The minimum effective percentages of the additive displaying the best protection was determined. The most effective additives were incorporated in minimum concentration in commercial greases which had failed to meet the rust preventive test to see if improvements could be made.

IMPROVING THE RUST PREVENTIVE PROPERTIES
OF MULTIPURPOSE GREASES

CONTENTS

IMPROVING THE RUST PREVENTIVE PROPERTIES OF MULTIPURPOSE GREASES

OBJECT

To improve the rust and corrosion preventive abilities of multipurpose greases by the use of additives.

INTRODUCTION

While the primary function of a grease is to lubricate and reduce friction, an equally important function is to protect the bearing surface from rusting and other forms of corrosion. Practically all current military, Federal and Industrial grease specifications contain some requirement concerning rust and corrosion prevention. Specification MIL-G-10924[1] Grease, Automotive and Artillery, is no exception and has always had some form of a corrosion and rust preventive test in all its revisions. MIL-G-10924A, the revision in use since 1956, contained two such tests. They were a copper corrosion test, Federal Standard No. 5309[2], and a synthetic sea water test based upon the Federal Standard salt spray test, No. 4001. Greases secured under the specification have successfully met these test requirements for a number of years.

Recently the Coordinating Research Council completed work on a rust preventive test method involving a Timken roller bearing cone and cup. Federal Standard Test Method No. 4012[2] was based on the CRC technique which was known as L-41. Since it was felt that this test more nearly approached actual use conditions, it was incorporated into the revision of the Specification MIL-G-10924B[1].

Suppliers of the MIL-G-10924 greases were somewhat concerned since the new test was admittedly more severe than those in use. Consequently this Laboratory was deluged with requests to suggest some additives which would enable a grease to meet the new requirements. Fortunately, enough preliminary work had been done so that two or three compounds could be suggested. It was deemed advisable to conduct a more exhaustive investigation to enlarge upon the list of possible additives and to determine what effect the successful ones would have upon other properties of the grease.

The various high molecular weight organic sulfonates and their metallic salts, lead soaps, amines, amine salts, naphthenates and non-ionic surfactants have been long regarded as rust preventives for oils and greases[3,4].

Baker, et.al.[5] have shown that of all the sulfonates, basic barium dinonyl naphthalene sulfonate in concentrations of 0.05% in petroleum oils and 0.5% in synthetic fluids gave the best corrosion inhibiting and acid deactivating properties.

Pines and Spivack[6] found that nonylphenoxy acetic acid protected ferrous metal surfaces from corrosion by adsorbing on the surfaces to form a close packed protective film. It is convenient to use for several reasons: (a) It is readily soluble in petroleum and synthetic fluids over a wide temperature range, (b) it is insoluble in water thus minimizing the tendency for the protective film to be desorbed by leaching into the aqueous phase, (c) its rust preventive properties persist in the presence of other additives and it appears to be compatible with most of them, (d) since it is a liquid, it can readily be adapted to a variety of blending technique, (e) it is organic in nature and consequently leaves little or no ash on combustion.

Preston[7] claims that compounds having the formula $R \cdot O \cdot CH_2 \cdot COOH$ where R is alkyl or alkaryl, and the alkyl groups preferably contain from 7 to 20 carbon atoms, are effective inhibitors of rusting when used in petroleum fluids. He found para nonyl phenoxy acetic acid to be the most effective of the compounds corresponding to the above formula. Concentrations of 0.02% enabled isooctane to pass the corrosion tests of MIL-I-25017A[8]. It was assumed that greater concentrations would be necessary for oils or greases.

The Armour Research Foundation working under contract with Wright Air Development Center screened several hundred compounds for their corrosion or rust preventive ability[9,10,11]. They also synthesized a number of compounds having potential corrosion inhibiting ability. Their work was with oils only and no greases were tested. The compounds evaluated in this investigation were chosen from those listed in these reports and from technical bulletins of various companies or from stocks available in this laboratory which were judged to be potentially successful.

PROCEDURE

One-hundred pounds of a multipurpose grease conforming to Specification MIL-G-10924A[1] was obtained to serve as the base grease. Eighteen likely additives were selected from those mentioned in the literature or which were considered as likely to be effective. They are listed in Table I. The additive was mixed into the base grease by means of a spatula. The percentages used varied somewhat and are listed in the pertinent tables. For preliminary evaluation a screening

TABLE I

ADDITIVES USED IN INVESTIGATION

1. Basic Barium Dinonyl Naphthalene Sulfonate
2. Sorbitan Monoleate
3. Para-Nonylphenoxyacetic Acid 90%
4. Stearoyl Sarcosine and Heptadecenyl Imidazoline
5. Undecyl Imidazoline and Oleic Acid
6. Morpholine
7. Lauroyl Iminodiacetic Acid
8. Dioctyldiphenylamine
9. Organic Sulfonates (commercial product)
10. Cadmium Diamyldithiocarbamate
11. Lead Dinonylnaphthalene Sulfonate
12. Lead Diamyldithiocarbamate
13. Commercial Lead-Sulfur E.P. Additive
14. Barium Dinonyl Naphthalene Sulfonate
15. Ethylene Diamine Dinonyl Naphthalene Sulfonate
16. Ammonium Dinonyl Naphthalene Sulfonate
17. Fatty Amido Phosphate
18. Anhydrous Lanolin

test was employed. This test took considerably less time and was more economical of materials than the specification test. It was conducted as follows:

Discs of #52100 bearing steel 1-3/8" in diameter, 5/16" thick and weighing 56.45 ± 0.05 grams were prepared. One surface of each disc was ground and finished with a diamond polishing wheel to obtain a finish equivalent to that of a bearing race. This surface was cleaned with xylene and air dried. As soon as the surface was dry, the additive-containing grease was spread over the surface with a stainless steel spatula. The excess grease was scraped off with the spatula and the disk placed grease side down upon a clean Kleenex tissue paper laid on a flat surface. The disc was then slid from one end of the tissue to the other with no downward force upon it except its own weight. The disc was then picked up and rotated approximately 90° horizontally, placed on a clean surface, and again pushed the full length of the tissue. The previously numbered discs were placed grease side up in a desiccator containing, in place of the desiccant, deionized distilled water. Upon each disc was placed six drops of deionized distilled water in a circular arrangement. The cover was placed on the desiccator which was then placed in a dark cabinet. The discs were checked at fifteen minute intervals for the first hour and then at hourly intervals thereafter. The time for appearance of rust was noted and when a very slight rust was noticeable on the last disc the test was discontinued. The discs were then given a numberical rating from 1 to 6 according to the severity of rusting.

The preliminary screening tests resulted in the elimination of additives No. 2, 4, 5, 6, 7, 8, 9, 10, 12, 13 and 14 from further consideration. Those additives which were most effective in the screening test were then added to the base grease and tested according to Federal Standard Test Method No. 4012. The base grease was also tested. Different concentrations of the additives were used in an attempt to determine the minimum concentrations which would provide sufficient protection. The additive-containing greases were also checked against the requirements of MIL-G-10924B to see if other properties of the grease had been altered.

Several of the most successful additions were also added to samples of commercial greases, including molybdenum disulfide containing greases, to further test their rust preventing abilities.

RESULTS AND DISCUSSION

One of the problems confronting the grease maker is the effect of any additive upon the over-all character of the grease. Some additives will affect other properties of a grease in addition to the one they were expected to enhance. Table II contains the results of several pertinent tests of Specification MIL-G-10924B upon the base grease and the base grease with two of the successful additives incorporated in it.

TABLE II

EFFECTS OF ADDITIVES UPON BASE GREASE

Property	Base Grease	+ Additive #1[1]	+ Additive #3[2]
Worked Penetration	266	286	271
Work Stability	+15	+28	+27
Water Stability	+38	+44	+12
Water Washout, %	4.57	3.9	5.2
Apparent Viscosity, Poises			
@ 25 $sec.^{-1}$	15,600	17,300	20,200
@ 100 $sec.^{-1}$	9,700	9,900	10,000
Copper Corrosion	Pass	Pass	Some darkening of grease

(1) Additive #1: 3% Basic Barium Dinonyl Naphthalene Sulfonate.

(2) Additive #3: 1% Para Nonyl Phenoxyacetic Acid.

Examination of Table II discloses that both additives did affect other properties of the grease. Additive #1, basic barium dinonylnaphthalene sulfonate, had an effect upon the worked penetration, work stability and apparent viscosity. Additive #3, para-nonylphenoxyacetic acid, affected the work and water stability and the apparent viscosity.

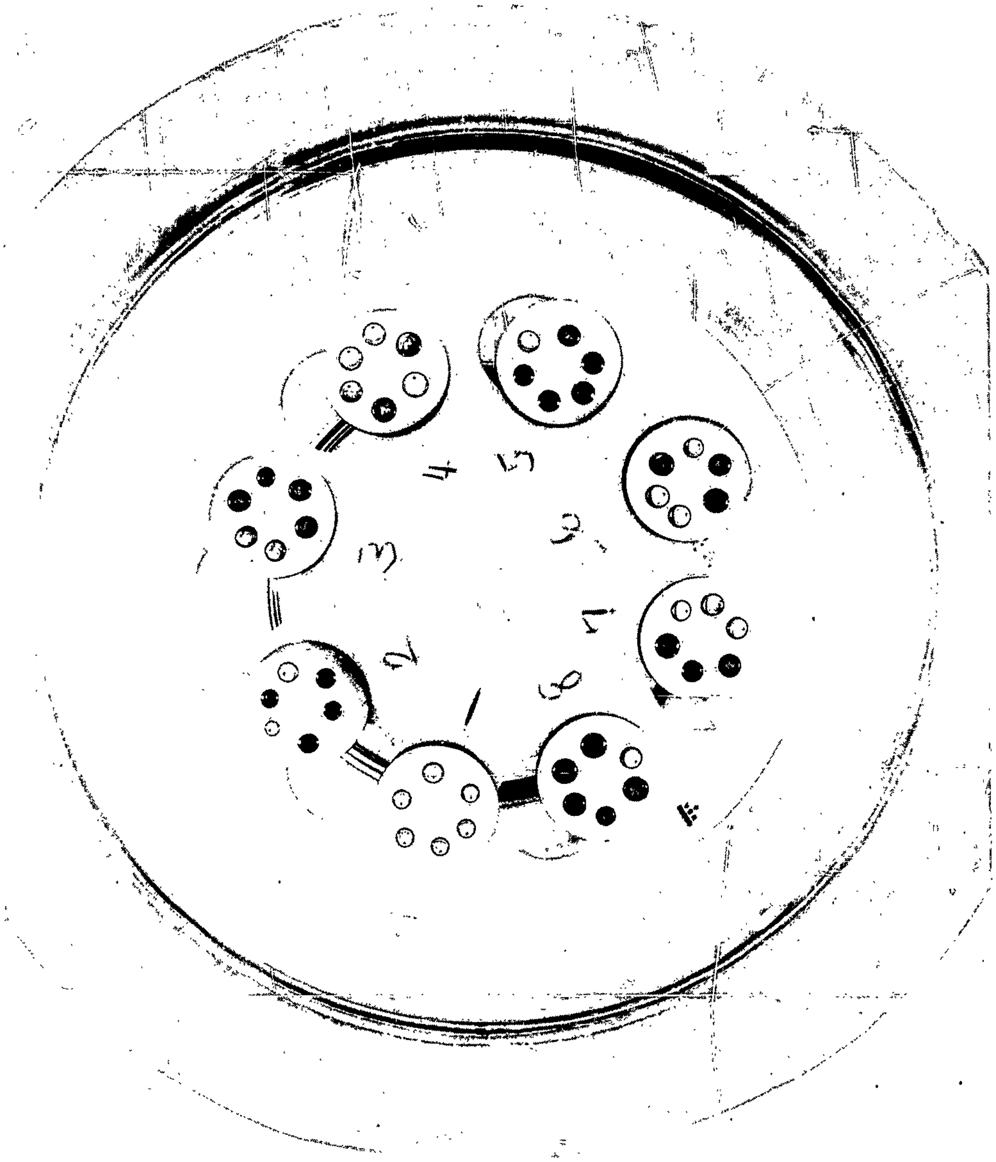
The results of the screening test made on a few of the compounds tested are shown in Figures 1 and 2 and in Table III. The ratings are based on the number of rust spots at the end of 72 hours or if less than 72 hours, were required the number of rust spots at the time all discs showed at lease one rust spot.

Figure 1 shows the discs in the desiccator at the conclusion of 72 hours. In Figure 2 the discs are lined up in order for better comparison. Table III lists the disc No., the number of the chosen additive taken from Table I, the additive percent, the number of rust spots at hourly intervals and the results of Federal Standard No. 4012 tests on the same grease-additive mixture.

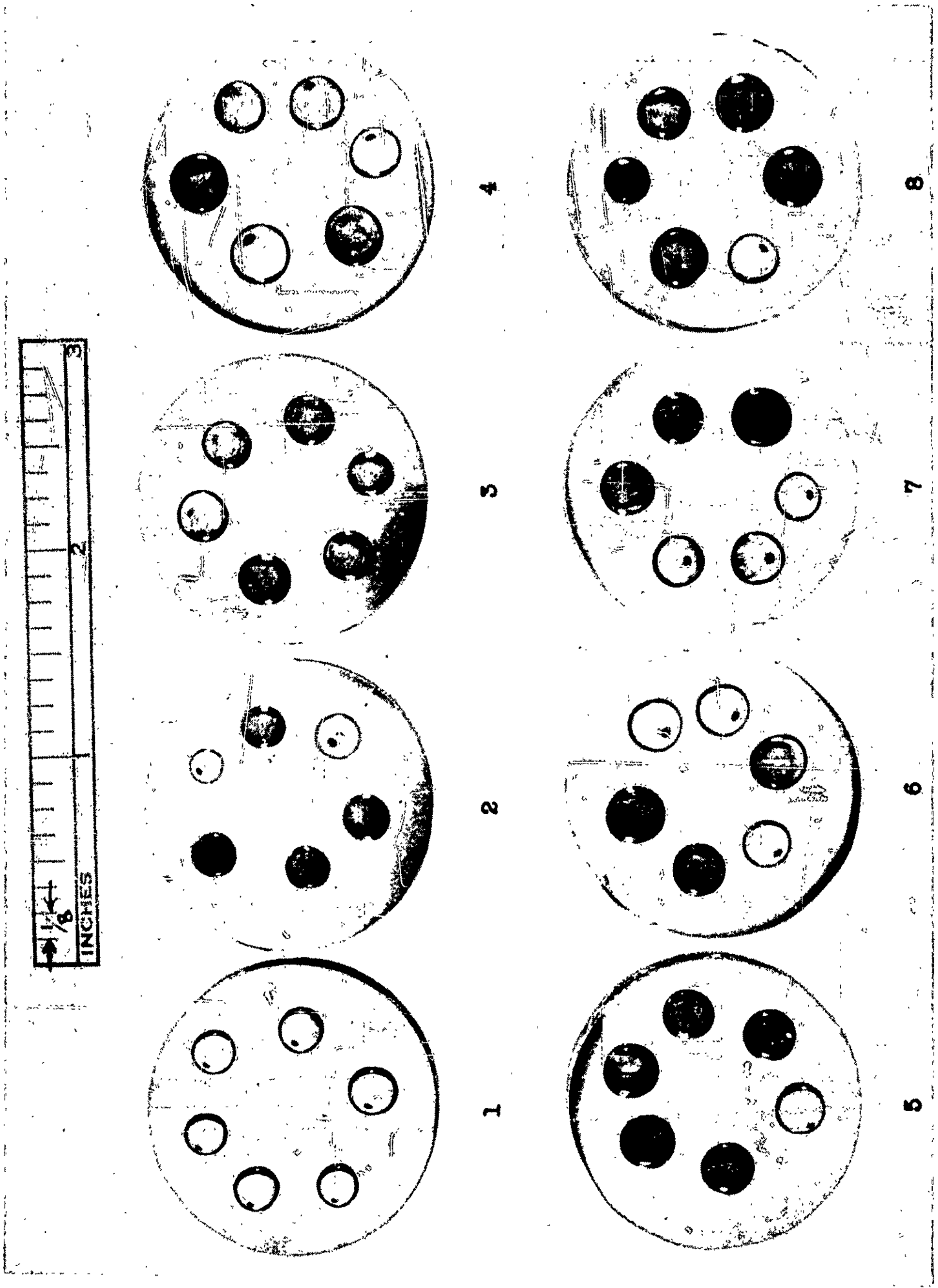
Examination of the data in Table III reveals that the results on the screening test are evident after a 4-6 hour period and are not significantly changed afterwards. Also, the screening test results are not very well correlated with the results of the Federal Standard No. 4012 test, as illustrated with Additives #1, 11 and 16. Since the 4012 technique has been standardized, and found to give reproducible results, it is considered to be the final answer in this investigation. The 4012 tests shown in Table III were made on two instead of three bearings for economy reasons. They are also shown pictorially in Figures 3 and 4.

To further test the effectiveness of these additives, two of them were tested in commercial greases as follows. The selected two additives were incorporated in a lithium soap W.P. grease designed for automotive use. Figure 5 shows the results of the Federal Standard 4012 test on the base grease and on the same grease containing the indicated amount of each additive. The additive containing greases successfully resisted rusting.

Figure 6 shows the results when 1% of nonylphenoxyacetic acid was added to a commercial molybdenum disulfide grease. The additive successfully controlled the rusting evident in the base grease.



SCREENING TEST FOR ADDITIVES IN GREASE

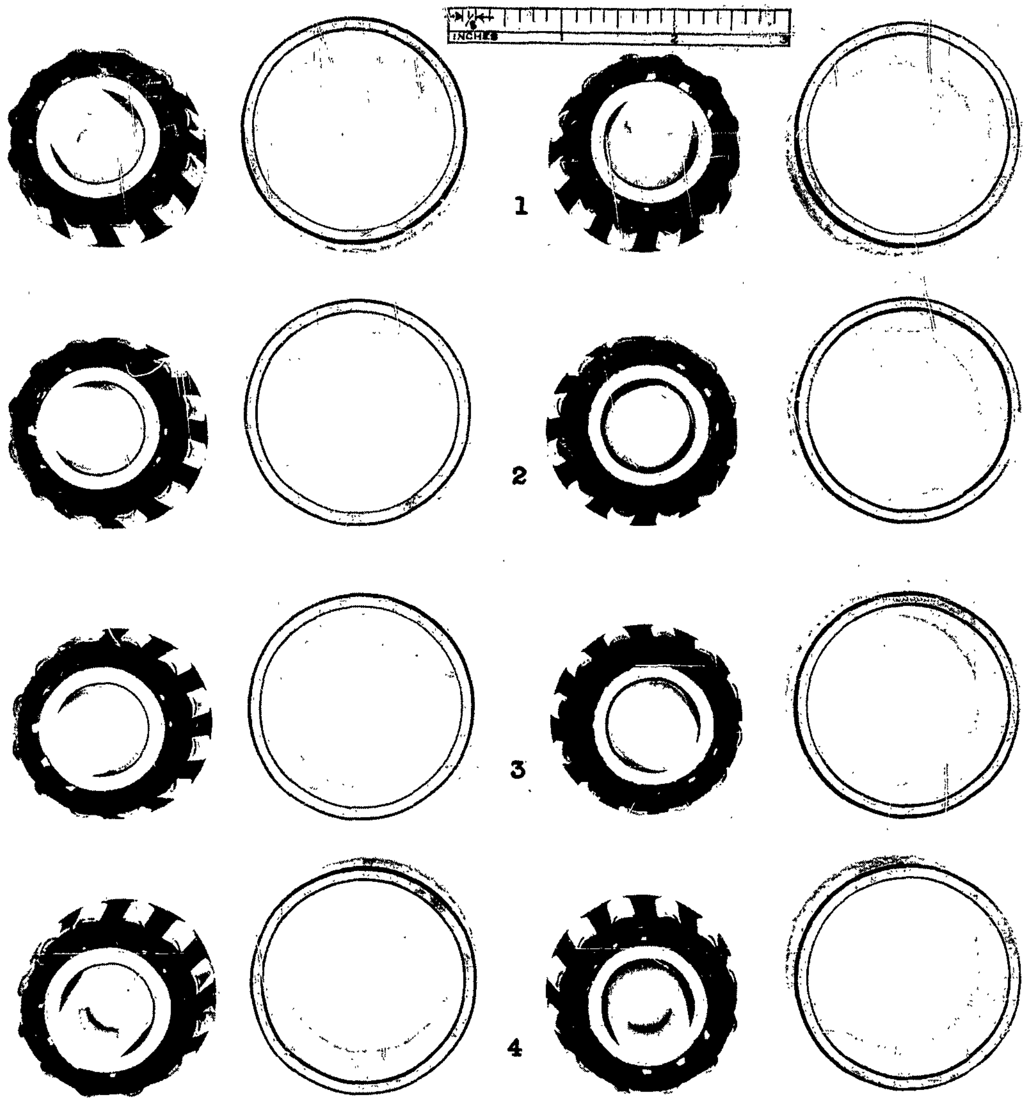


SCREENING TEST FOR ADDITIVES IN GREASE

TABLE III

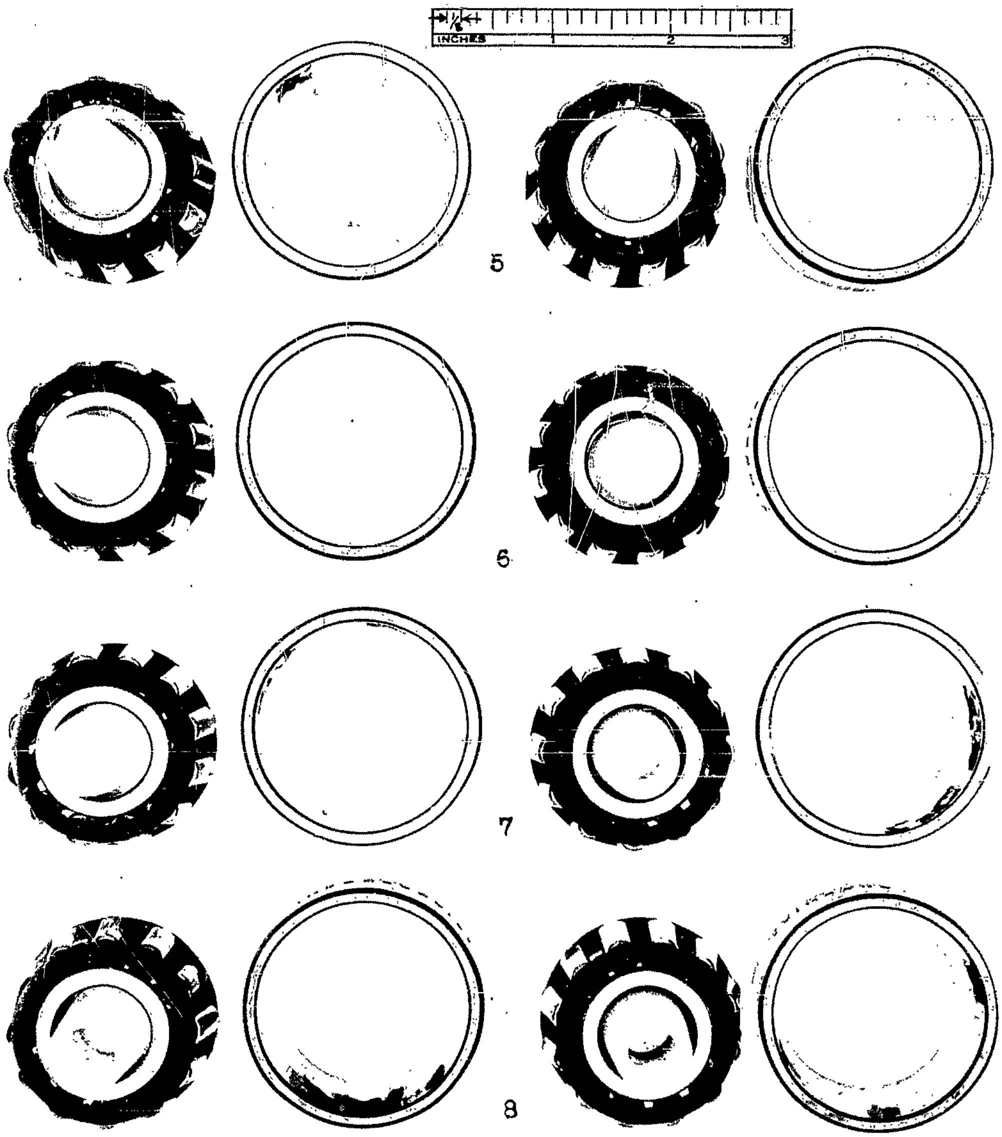
SCREENING TESTS ON ANTIRUST ADDITIVES

Disc No.	Additive No. From Table I	Additive Percent	Rust Spots on Disc at Time Indicated							Results Fed. Std. No. 4012 Test
			1 hr.	2 hr.	3 hr.	4 hr.	5 hr.	6 hr.	72 hr.	
1	3	1	0	0	0	0	0	0+	1	1,1
2	11	1	0	3	4	4	4	4	4	1,1
3	16	1	0	0	4	6	6	6	6	1,1
4	15	1	0	0	2	3	3	3	3	1,1
5	1	1	0	4	4	5	5	5	5	1,3
6	17	2	0	2	2	3	3	3	3	1,1
7	18	2	0	3	3	3	3	4-5	5	3,3
8	Base Grease		0	2	5	5	5	5	5	3,3



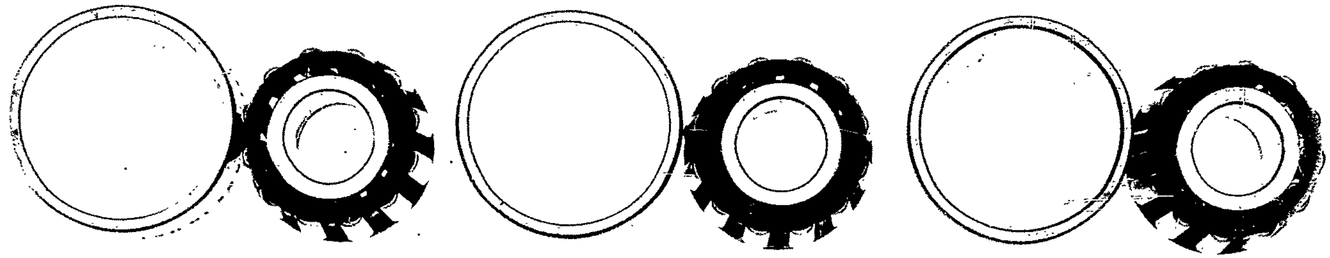
FEDERAL STANDARD 4012 TEST METHOD ON VARIOUS
ADDITIVES IN A MIL-G-10924A BASE GREASE

FIGURE 3

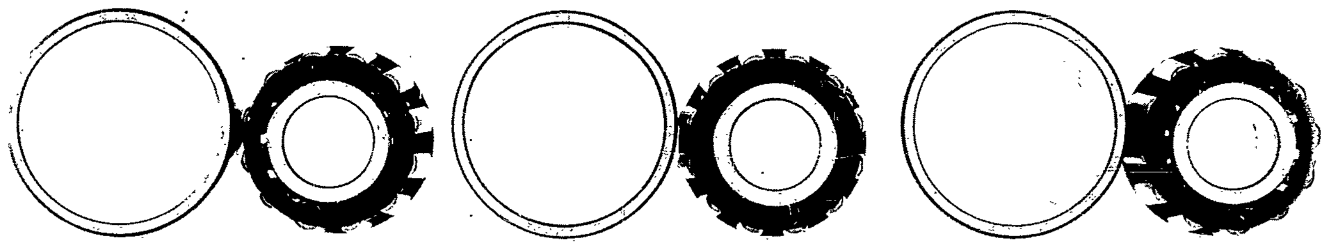


FEDERAL STANDARD 4012 TEST METHOD ON VARIOUS
ADDITIVES IN A MIL-G-10924A BASE GREASE

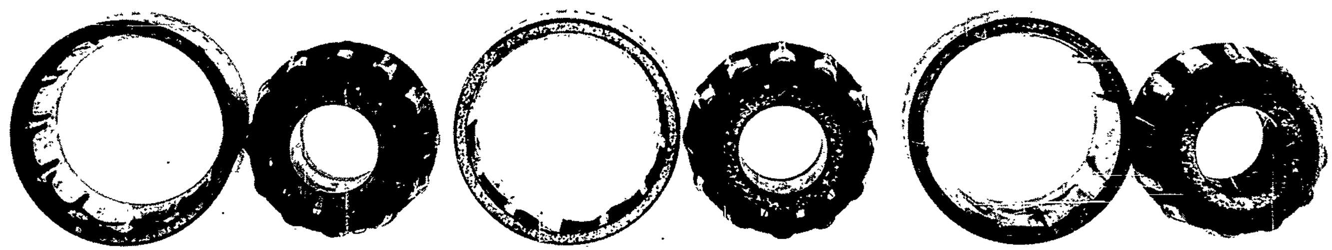
FIGURE 4



1½% NONYL PHENOXY ACETIC ACID IN E.P. LITHIUM GREASE

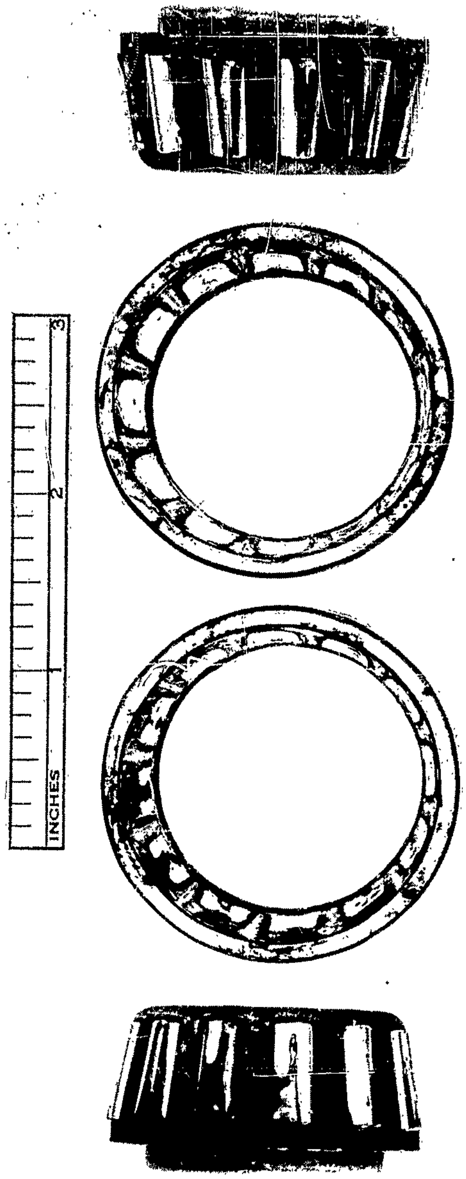


3% BASIC BARIUM DINONYL NAPHTHALENE SULFONATE IN E.P. LITHIUM GREASE

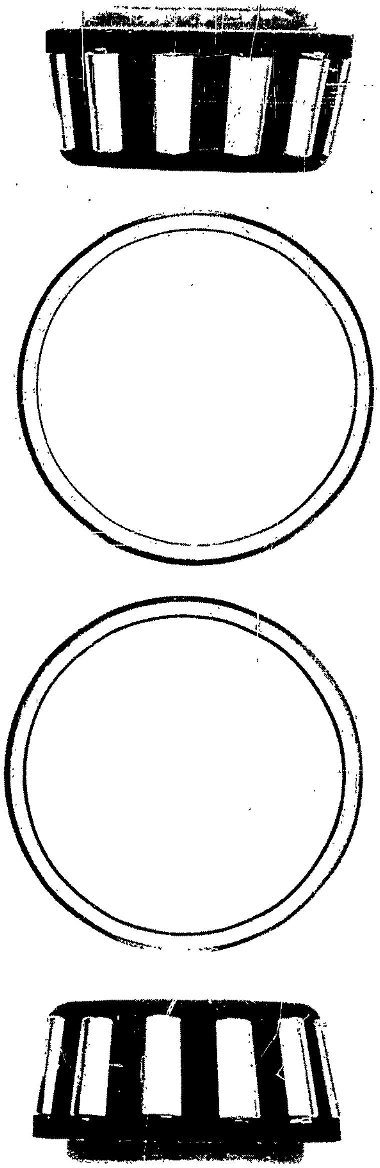


E.P. LITHIUM GREASE

FEDERAL STANDARD 4012 TEST; RUST PREVENTIVE PROPERTIES OF GREASES



RESULTS OF FED. STD. 4012 ON MoS_2 GREASE.



RESULTS ON SAME GREASE + 1% NPA.

FIGURE 6

SUMMARY

1. The rust preventive properties of multipurpose greases can be improved by the addition of relatively small amounts of several different compounds.

2. These compounds are also effective in other types of commercial greases.

3. Other important properties of the greases were also affected by the additives and care must be taken to allow for this in the final formulations.

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